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	SUMMARY REPORT	
	PERIOD: NOVEMBER 1, 1971 to NOVEMBER 30, 1971	
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1.0 CHEMICAL R and D

Materials' Purification and Synthesis

D260:

The first two hundred grams of D260 were received from ChemSampCo (Columbus, Ohio) during the early part of The material was found unsuitable for photograde purification! Meanwhile, additional D260 was prepared in-house and also found unsuitable for photograde purification! A close examination of this problem reveals, that in all probability, purity of the intermediate Michler's Hydrol is tantamount to obtaining good D260. Fortunately, ChemSamCo had converted only a small portion of the other key intermediate so that the bulk of D260 will now be prepared using purified Michler's Hydrol. ChemSampCo's purification of the hydrol will cost an additional \$300. Additional D260 is also being prepared in-house in order to secure more D260 at the earliest possible date and also to stop-gap any additional complications which might be encountered by ChemSampCo.

Unfortunately, the current stock pile of D260 which was allocated to pilot coating has been depleted and only a minimal amount remains for sustaining the research effort. It is estimated that sufficient D260 will be available for resuming and sustaining the pilot coating effort no later than December 13. Meanwhile attempts are also being made to find suitable adjustments in the photograde purification methods to convert the existing, unsuitable material to photoquality. If this succeeds, suitable D260 will be available well before the 13th.

CBr₄:

Preliminary analytical data from "good" and "bad" CBr_4 indicate that the impurity which was discussed last month is hexabromoethane, $\mathrm{C_2Br}_6$. Conclusive analytical data is expected by the end of December. New evidence suggests that hexabromoethane is not detrimental to $5/\mathrm{D7}$'s photoproperties. If this is so, and the detectable impurity does prove to be $\mathrm{C_2Br}_6$ then last month's conclusion, that this impurity is directly responsive for film failure, is incorrect and

another trace impurity must be sought. To support this evidence authentic, purified C₂Br₆ is being prepared for film evaluation and professional analytical work continues to attempt identification of other impurities, particularly oxygen containing bromine compounds which are firmly suspected as the real cause of the CBr₄ problem.

An effort is currently underway to contact all prime produces of CBr₄ (worldwide) and procure samples of their product.

Some development work was done on the preparation of CBr_4 by the haloform reaction on acetone, to make laboratory preparation by this alternate synthesis route more practical. The literature route requires addition of small portions of the reactants separated by time intervals. Only a small loss in yield resulted from an experiment where all of the reactants were added at once and stirred overnight. An eight-fold scale-up of this was also practical, so that it is now possible to run in a 12-liter flask and produce 112 g CBr_4 per day with little attention required. A scale-up with a reduced volume of water gave very poor yield.

An attempted synthesis of carbonyl bromide, COBr₂, (a suspected impurity) by the oxidation of carbon tetrabromide with concentrated sulfuric acid led to a crude material containing a large concentration of bromine. By the time the bromine was removed by chemical action of ethylene and mercury the carbonyl bromide had apparently been destroyed and/or volatilized. The partly purified crude was apparently a mixture of ethylene bromide and tribromoacetyl bromide. Identification of the latter was by comparison of its anilide, tribromoacetanilide, CBr₃CONHC₆H₅, which was identical by infrared spectrum to tribromoacetanilide prepared from authentic tribromoacetyl bromide. No further work is planned on the preparation of carbonyl bromide.

4P0:

Additional photograde 4PO was prepared and stockpiled to forestall a shortage which would affect pilot coating operations.

Polystyrene Binder:

Subsequent to last month's discussion, the decision was made to postpone anticipated complications of replacing the experimental MX4500 (used exclusively on P325 and P325A) by placing a special minimum order for 10,000 pounds from This decision was based on preliminary film Dow Chemical. results which showed that several commercial grade polystyrenes (Borden's 230,000, Lestrex, Styron) were no better and perhaps much poorer (insufficient data) than MX4500 in their effects on photographic properties. It was deemed undesirable to compound existing work loads at this time in an effort to find a suitable replacement, and furthermore, all research data to date has been compiled using MX4500.

Unfortunately, it has recently been learned that Dow has decided to discontinue MX4500 and will not produce it even on special order. Furthermore, Dow indicated that no existing supply remains.

Dow has supplied a sample of Styron 685, a "high heat, general purpose polystyrene" which it indicates is, for all practical purposes, identical to MX4500. Immediate evaluation of Styron 685 is scheduled and it is hoped that it will be a suitable replacement, or otherwise a more extensive screening program will be necessary, at a time when pilot coating and shelf-life are paramount problem areas.

New Materials:

Samples of 2PO, 3PO, 2,6-dichloropyridine-N-oxide, 3-hydroxypyridine-N-oxide and 4-nitropyridine-N-oxide have been procured and purified. Film evaluation will be made for comparison to 4PO particularly in relation to effects on shelf-life/speed decay.

Two azo dyes are being prepared as representative members of a hitherto uninvestigated class of dyes which might be suitable as spectral sensitizers. The preparation of one of these, 4-2-naphthalen-azonaphthylamino-1, has already been completed.

Film Studies

Ethylene Dichloride Solvent Systems:

Last month's pilot coating work had led to the conclusion that benzene is a poor solvent for meniscus coating techniques. A placebo, analogous to 5/D7 had also indicated high quality machine coatings were possible with 1,2-dichloroethane. As a result of that work the first part of November witnessed an effort to marry the favorable coating properties of dichloroethane (EDC) to the photographic properties exhibited by the 5/D7 (benzene) system. This effort and its results will be discussed here and the resultant machine coatings will be discussed in greater detail in the Pilot Coating section.

The laboratory research effort began with a direct replacement of benzene with dichloroethane (EDC) which resulted in a premature dark reaction to give fog (the usual magenta). This contrasts with the benzene system which has never been observed to fog even after days of ambient storage. Removal of D7 failed to prevent the fog which was now blue (D392 the corresponding dye of D260). This observation showed that fogging was not due to acid present in or formed by EDC, since D260 does not respond to acid. It also indicated that the cause of fogging was in all probability a free radical oxidation reaction. (If this is so it is worth noting that not only might EDC being favoring or causing the reaction, but rather that benzene might be inhibiting an otherwise inherent reaction).

The next obvious step was to add an antioxidant which would prevent the unwanted, fog-producing reaction(s), without seriously hindering the normal, light induced, imageforming reactions. Plastinox 425 was chosen since past work with Formula 1 showed that of those few antioxidants which had been investigated it was the least deleterious to image formation. It was found that 1.69 mg was the minimum amount necessary to prevent the premature fogging. level had virtually no effect on the printout speed of 5/D7 (compare Figures 1 and 2), but a significant retardation was observed in the development mode. Higher levels of the Plastinox almost completely destroyed the developability. Six other additives: Plastinox 2246, 2,6-di-tert-butylphenol, triphenylstibene, 2,4-di-t-amylphenol, Santonox R and Santowhite (Monsanto), were also tried. All failed in various degrees to match the effectiveness of Plastinox 425.

At this point it was decided to continue this approach. Thus, the printout mode was virtually uneffected and the development mode, though severely retarded, was still operative. The 5/D7(EDC Plastinox) system thus afforded the means of demonstrating the machine coatability of the basic P-325A material while retaining at least a portion of its sensitometric capability.

Up to this point the EDC had been stabilized with a trace of triethylamine (TEA). Past experience with this solvent and Type 2000 duplicating film had demonstrated that this stabilization was necessary to prevent the buildup of trace acid impurities. The question was what effect TEA had on the photographic properties of 5/D7(EDC, Plastinox). Subsequent experimentation demonstrated that the level of TEA used for stabilizing EDC had no observable effect on photographic properties. Stabilized EDC was therefore chosen for continued use.

The last step prior to a trial machine coating was to determine if the, then optimum, concentrations of 4PO and CBr_4 had been altered by the addition of Plastinox 425 or by perhaps even the EDC. Independent traversals of both ingredients showed the same concentrations as still optimum.

The final formulation was therefore established as identical to 5/D7(benzene) but with EDC in place of benzene plus 1.69 mg of Plastinox 425: 5/D7(EDC, 1.69 Plx 425). Several machine coatings (325014, 015 and 016) were made with the results being fairly uniform coatings and a printout exposure comparable to that obtained in the laboratory (see Figure 3). In contrast to last month's runs with the benzene system where oven drying at 110°F caused total fogging; these runs, also dryed at 110°F, produced at most only 0.1 - 0.3 units of fog density. This had been expected since laboratory work showed this improved thermal stability for the EDC system, most probably as a result of the antioxidant, Plastinox 425. These runs, however, still produced tacky film at 110°F so temperature was raised to 150° and 200°F for the second and third zones of the oven. These temperatures produced tackless films but caused total fogging. At this point work returned to the laboratory to see if higher levels of Plastinox 425 would permit these higher drying temperatures.

A traversal of higher Plastinox 425 levels showed that 6.75 mg would prevent serious thermal fogging at 150°F but not 200°F. Higher levels of Plastinox virtually destroyed photosensitivity. Reformulation was therefore set at 6.75

.25X1

mg of Plastinox with a maximum drying temperature of 150°F. Under these conditions net densities were significantly lower than the 5/D7(benzene) and 5/D7(EDC, 1.69 Plx 425) systems. Developable sensitivity was almost completely destroyed with several faint steps being barely discernible. This formulation, 5/D7(EDC, 6.75 Plx 425) was machine coated with drying at 150°F to demonstrate the capability of machine coating this system to obtain tackless, uniform coatings while retaining at least some photosensitivity. Further discussion of these results can be found in the Pilot Coating section.

Additional laboratory studies with the 5/D7(EDC, 1.69 Plx 425) system led to another interesting observation. The enhanced thermal stability of the EDC system led to the obvious question concerning whether the shelf-life/speed decay had also been improved. A series of comparative ambient temperature speed decay measurements was made between the 5/D7(benzene) and 5/D7(EDC, 1.69 Plx 425) systems. The printout mode was used since both systems exhibited virtually identical results initially (compare Figure 1 and 2). After 5 hours the densities of the benzene system had decayed more than for the EDC system, but the EDC system had developed fog while the benzene system had not. This represents another element in a series of accumulating observations which indicate that more than one mechanism is operating in the speed decay phenomenon.

Other Solvents

The apparent need for a solvent change prompted an
investigation of still other solvents. To be selective
and still try several types, the solvents were chosen based
on the experiences of the Production Manager of
He had recommended these solvents as being
suitable for meniscus coating of polystyrene. In addition,
specific structural types of chlorinated hydrocarbons were
chosen to determine if any structure/activity relationships
exist. Table 1 summarizes the results of these experiments.
All but one either failed to dissolve the photoingredients
and/or caused fogging. The single exception was tetra-
chloroethylene (perchloroethylene, perchlene) which although
exhibiting some solubility problem gave developable photo-
response similar in appearance to that exhibited by the
5/D7(benzene) system. This was not surprising since this

25X1 25X1 solvent was predicted as being the probable choice. This prediction was based on the fact that it contains no hydrogen which could result in acid formation or support a free radical chain reaction and the presence of a double bond which could mimic benzene in its ability to complex CBr₄. Another interesting observation was the apparent absence of "orange-peel" (broken surface; appearance of orange skin or record grooves) with tetrachloroethylene, which could be an alternate approach to the use of silicone oil for the elimination of orange-peel. Some work has continued with tetrachloroethylene, including mixtures with benzene. The results will appear in a future report.

Pilot Coating

Last month's report described the poor coating quality obtained with the 5/D7(benzene) system and offered an explanation based upon the unacceptable role played by benzene. To illustrate this point a coating was made using ethylene dichloride (EDC) as solvent, and the result was a good quality coating comparable to the best qualities obtained with Types 2000 and 2500.

These results led to a research effort to replace benzene with EDC in the 5/D7 system. This effort and its results have already been described under Film Studies. A concerted effort was made during the early part of November to machine coat the 5/D7(EDC) systems.

In order to conserve valuable and limited supplies of photograde materials, especially D260, a liberal use is being made of machine coated placebos. In general, the placebo employs D290 for D260, a cresol for D7 and 4PO, and crude or purified but photographically unacceptable CBr₄. In this way formulation and coating parameters can be determined or adjusted without consuming the currently limited supplies of photograde materials.

The results of the machine coated 5/D7(EDC) systems are summarized in Table 2.

The first run (325014) contained no DC510 silicone oil. The result was severe "orange-peel" and illustrates the need for a surface-active agent such as DC510.

DC510 was added to the remaining bulk of coating solution and Run 325015 then made. The coating pattern was fair while photosensitivity was evidenced by a measureable printout (AEI 1.6 x 10-4). The speed and densities of this printout (Figure 3) were comparable to the 5/D7(benzene) and the 5/D7 (EDC)systems that were hand coated at ambient temperatures (Figures 1 and 2) but the fog level was considerably higher as a result of the higher drying temperature (110°F).

Since Run 325015 showed some "face-to-face" tack, the remainder of the coating solution was coated with a drying temperature of 150 to 200°F (Run 325016). The result was a tackless but totally fogged coating. The fog resulted from the higher temperatures as proved by subsequent laboratory experiments, and was probably further compounded by the age of the coating solution, then about 1-1/2 hours old.

The results of Runs 325014 and 016 led to additional laboratory experiments on the effects of temperature which in turn culminated in Run 325017. This run employed the same basic formulation but with additional antioxidant (Plastinox 425) to compensate for the higher drying temperature of 150°F. The result was the highest quality coating yet obtained and with but a slight face-to-face tack. The coating possessed photosensitivity (AEI 3.6 x 10-5, Figure 4) to the printout mode but showed only a barely detectable response to develop-The lower printout speed for this run was expected on the basis of laboratory experiments and is due to the higher levels of antioxidant. Cold storage (-30°C) for 72 hours resulted in retained photosensitivity as evidenced by a printout AEI of 7.9 x 10^{-5} . Surprisingly, the aged film showed a significant increase in density with no increase in fog (Figure 5).

To put into perspective the significance of these AEI printout speeds, an exposure was made to the Military Systems Department's stationary printer, the same being used for evaluating Type 2000 films. The result was a speed of 15 mj for a net density of 1.0 (Figure 6), which is approximately three times the sensitivity of Type 2000. An example of this exposure is in the possession of the customer.

The above work and results were described to the customer in an oral briefing presented in his office on November 9, 1971. At this point the results were far short of the desired

objective for 1971, but they did represent a significant step forward in the efforts to machine coat P325A material. Thus, a very important fact had been established: that 5/D7, the prime system of P325A, can be machine coated to give quality as good as anything yet obtained with the Talboys coater while retaining a significant part of the inherent, primary sensitivity. It should be remembered however that only short lengths of film are produced, 6' - 10' in length, and this material is tacky! Having thus demonstrated the ability to machine coat the 5/D7 system, efforts were directed toward the ultimate objective of machine coating with a retained sensitivity of AEI > 1.6.

Subsequent to the customer's briefing of November 9, it was decided to reinvestigate the benzene system as the logical starting point for the continued effort. This decision was based upon a strong suspicion that the work described in last month's report had been misleading, and that the explanation based on the properties of benzene was inconsistent Thus, it had been explained that the with certain facts. observed behavior of the benzene system was akin to that of systems employing methylene chloride as solvent, and hence its volatility is too great for the current limitations of the Talboys coater. In contrast to this explanation, published data on comparative volatilization rates of solvents show that in fact the volatility of benzene is only slightly greater than that of EDC while being considerably less than that of methylene chloride.

The renewed effort began with Runs 325018 and 019. These were the usual placebos for the 5/D7 system and employed benzene as solvent; excellent coatings resulted! Coating personnel reported that the wet, freshly coated film did not "errupt into an extremely turbulent discontinuity of liquid" as had been observed and reported last month. Numerous runs, both placebos and 5/D7, have since been made and not a single instance of that first described behavior has been encountered.

Prior to the actual coating of 5/D7 a number of additional placebos were run. Several of these placebos actually included all the photoquality ingredients of 5/D7 except D260. In this manner the effects of D7 and 4PO, the least soluble ingredients in the formulation, could be determined without risking the critical supply of photograde D260. The results of all these preliminary experiments and placebos indicated that the

5/D7(benzene) system should machine coat without difficulty.

The coatings of the 5/D7(benzene) system began on November 23 with Runs 325040 and 041. These runs were prefaced with a concerted effort between the coating and research personnel to plan every step of the experiment so that no personnel to plan every step of the experiment so that no time would be lost. Time is currently a critical factor since the shelf-life/speed decay properties confine the desired, higher sensitivities to the first half-hour after coating. Exposure and development equipment was stationed in a room opposite the coating alley. The first few feet to be coated could then be cut and immediately transferred to this room for evaluation. In the meantime, the remaining thirty or so feet could be coated, then cut and packaged for refrigeration since laboratory experiments have indicated that three weeks of shelf-life can be preserved at -30°C.

The results of the three runs made during November are summarized in Table 3.

The first run, 325040, produced a poor coating similar to that obtained in October. The problem was quickly assessed to be due to an unexplainable shortage of DC510 silicone oil (placebos had indicated that the original amount of DC510 should have been sufficient). Additional DC510 was added and Run 325041 was made. The result was a tacky, good quality, coating. The printout speed was appropriate to high speed film and a developable speed of AEI 0.10 was obtained.

The following day a repeat, Run 325042, was made; this time with the higher level of DC510. The result was a good quality coating with AEI speeds greater than 0.10. Three hours of refrigeration showed retained sensitivity. With this run the existing supply of photograde D260 had been depleted, and this problem has already been discussed with respect to D260. Additional supplies of photograde D260 are expected no later than December 13 or sooner.

In any event, Runs 325041 and 042 indicate that speeds of 1.6 or greater should be attained before the end of the year. There are several reasons for this statement.

First, the speeds of 0.10 to 0.17 were obtained in a room too cold (60°C) for optimum results. Laboratory experiments in the past provide ample evidence that a temperature differential of $10^{\circ} F$ ($70^{\circ} C$ about optimum) can easily account for a ten-fold difference in speed.

Secondly, the speeds of 0.10 - 0.17 were limited by the onset of blotch as always, but in these instances the blotch followed a pattern definitely attributable to variations in coating thickness. It is thought that these thickness variations were due to viscosity variations within the bulk coating solution resulting from improper handling. If this is so then appropriate corrections in handling procedures can be made. An alternate possibility exists, however. It is possible that the coating quality is as good as currently possible with the Talboys, and not good enough for high speed Thus, it is conceivable that the 5/D7 system itself, because of the phenomenon known as blotching, is the most sensitive measure of coating thickness variations.

-11-

PROBLEMS

Chemical Samples Company's delivery of poor quality D260 prime dye materials.

FINANCIAL

Approval received for funds to cover the present 90-day effort.

PLANS FOR NEXT REPORTING PERIOD

- 1. Produce additional trial coatings of full formula 325A layers.
- 2. Analyze results and bear on problems associated with trial coatings.
- 3. Preview engineering and chemical work for follow-on after January 1st.

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TABLE 1
SOLVENT REPLACEMENTS FOR BENZENE (5/D7)

Solvent	Solubility*	Fogging	Photo- Sensitivity	Developable Imaging	J
					-
Ethyl acetate	No	No	No	4	
n-Butyl acetate	No	Light (grey)	No	÷	
Methyl ethyl ketone	No	Heavy (blue)	'_		
Methyl i-butyl ketone	No	Moderate (purple)	-	•	
Tetrahydrofuran (THF)	OK	Heavy (purple)	_		
1,4-dioxane	Poor	Light (purple)	-		
Trichloroethylene	OK	Very light (purple)	Yes		
Tetrachloroethylene	Fair	No	Yes		
1,1,1-trichloroethane	OK	Very light (purple)	Yes		
Ethylene dichloride (EDC)	OK	Moderate (purple)	 '		2
Fluorobenzene	Poor	No	Yes	Fair	Ζ,
Toluene	OK	Very Slight (pink)	Yes	${f Poor}$	
Xylene	OK		Yes	Very Poor	

^{*} Refers to photoingredients - all dissolve polystyrene.

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25X1

TABLE 2
P325A PILOT COATING DATA

November, 1971

Ethylene Dichloride Solvent System¹: Formula 5/D7

Run	Plastinox Vi 425 (mg) ²	scosity (cps)	Coati Spee	ng O d #1	ven Zor #2	ne(°F) #3	Pattern	AEI (Printout)	Figure	Comments
325008	-(Placebo)	19.5	10	off	100	100	very good	-	-	face-to-face tack
325014	1.69	16.5	10	off	110	110	orange peel			no DC510
325015	1.69	16.5	10	off	110	110	fair	1.6x10 ⁻⁴	3	compare to Fig. 1 and
325018	1.69	10.0	15	off	150	200	-	· •	-	total thermal fog no tack
325017	6.75	10.5	15	off	150	150	very good	3.6x10-5	4	
								7.9x10 ⁻⁵ (72 hrs)	5	slight face-to-face tack
						-		ergs/1.0Dne	t 6	tack 25X1

amine (TEA) stabilized

standard 4.5 ml coating solution

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TABLE 3
P325A PILOT COATING DATA
November, 1971

Benzene Solvent Systems: Formula 5/D7

Run	Viscosity (cps)	Coating Speed (fpm)	Oven #1	Zone	e(°F) #3	Pattern	AEI (printout)		Figure)	Comments
325019 (and ot	12 thers)	17.2	off	off	50	very good			-	Placebo
325021 (and ot	9.25 thers)	17.2	off	. 50	50	very good	-	- -	-	Placebo*
325040	12	17.0	off	off	60	terrible! orange pee	- ,	-	-	unsufficent DC510
325041	12+	17.0	off	off	60	very good	6.4×10^{-4}	0.17	7 8	more DC510 0.30 mil dry thickness
325	2	17.0	off	off	60	fair	3.1x10 ⁻⁴	0.10	9 10	25X1
						·	1.5x10-4	0.16	11 12	{3 hrs. at -30°C
* A	grade	materia	ls of	5/D7	syste	m except D290	for D260			

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